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Determination of fiber orientation in Norway spruce using X-ray diffraction and laser scattering

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Abstract Spiral grain reduces the quality of timber since it causes twisting during drying and reduces the mechanical strength of wood products. The orientation of wood fibers in Norway spruce as a function of the distance from the pith was studied using both x-ray diffraction and light scattering. In radial-longitudinal plane upper tips of fibers were tilted towards the pith and the tilt angle increased gradually towards the bark in most of the samples. Periodic oscillations in the spiral grain angle were observed. Increased growth rate was found to increase the amplitude of this oscillation. There was no clear correlation between the angles determining the fiber orientation and other parameters like the lumen diameter, the cell wall thickness, the density of the sample, the fiber length, the circularity index of the cell lumen, or the mean microfibril angle. However, fiber orientation in tangential-longitudinal plane varied more in broad annual rings than in narrow annual rings.

Bestimmung der Faserneigung in Fichtenholz mittels Röntgenbeugung und Laserstreuung

Zusammenfassung Drehwuchs verringert die Holzqualität, weil er zu Verdrehungen beim Trocknen führt und die mechanische Festigkeit der Holzprodukte verringert. Mittels Röntgenbeugung und Laserstreuung wurde die Faserneigung in Fichtenholz in Abhängigkeit vom Abstand zur Markröhre untersucht. In radial-longitudinaler Ebene neigten sich die oberen Faserenden zum Mark hin. Zur Rinde hin stieg der Neigungswinkel bei den meisten Proben allmählich an. Der Faserwinkel unterlag periodischen Schwankungen, die mit zunehmender Jahringbreite grö-

ßer wurden. Eine eindeutige Korrelation zwischen den Faserwinkeln und anderen Parametern, wie zum Beispiel Zelllumen-durchmesser, Zellwanddicke, Probendichte, Faserlänge, Rundheitsindex der Zelllumen oder dem mittleren Mikrofibrillenwinkel, konnte nicht festgestellt werden. Die Faserneigung in tangential-longitudinaler Ebene variierte jedoch bei breiten Jahrringen mehr als bei schmalen Jahrringen.

1 Introduction

Wood material of coniferous trees consists mainly of spindle-shaped tracheids, which are aligned almost parallel to the longitudinal direction of the stem. Deviations from this direction, called spiral grain, can be found in every tree (Kozłowski and Winget 1963). The definitions used for deviations from the longitudinal direction, the angles α (radial deviation) and β

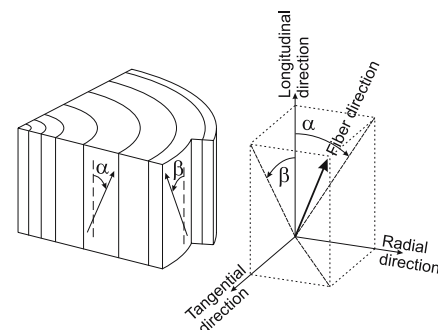


Fig. 1 Geometry of the measurements. Wood sections were cut in transverse direction from the pith to the bark at breast height (1.3 m) using double circular saw. The mean orientation of the fibers is defined by the angles α and β and the positive direction of the angles is marked with arrows. The angle between the longitudinal direction of the stem and the longitudinal direction of the fiber in radial sections is α . The angle β is spiral grain angle

Abb. 1 Messgeometrie. Mit einer Doppelkreissäge wurden Stammscheibenabschnitte in Brusthöhe (1,3 m) herausgeschnitten. Die Winkel α und β geben die mittlere Faserneigung an. Die Pfeile bezeichnen die positive Winkelrichtung. α ist der Winkel zwischen der Längsachse des Stamms und der Faserneigung in radialer Richtung. β bezeichnet den Drehwuchs

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(tangential deviation) are shown in Fig. 1. In most coniferous trees the fibers are left-handed spirals ($\beta > 0^\circ$) near the pith, but the fiber orientation changes to right-handed after 10–30 years (e.g. Kozłowski et al. 1967, Pape 1999, Gjerdrum et al. 2002).

The corkscrew-like spirality has a negative effect on the quality of wood, because it impedes sawing and other processing (Johansson et al. 2001, Kliger 2001, Perstorper et al. 2001, Johansson and Kliger 2002). It also reduces the strength of the sawn timber (Kollman and Côté 1984) and causes twists during drying. Even small changes ($\sim 5^\circ$) in the orientation of the fibers

have significant effect on the quality of the surface of plywood and rotary-cut veneer (Harris 1989).

The most widely used technique to determine spiral grain is probably the scribe test method (Northcott 1957). It involves forcing a sharp point through the wood surface and measuring the fiber orientation from the scratch. Typical accuracy of the measurement is 0.5 degrees.

Computeraided tomography (CT) has also been used to study spiral graining in centerboards of Norway spruce (Sepúlveda et al. 2003). The resolution was, however, quite low and the mean orientation of fibers was determined in 5 mm bands. Better reso-

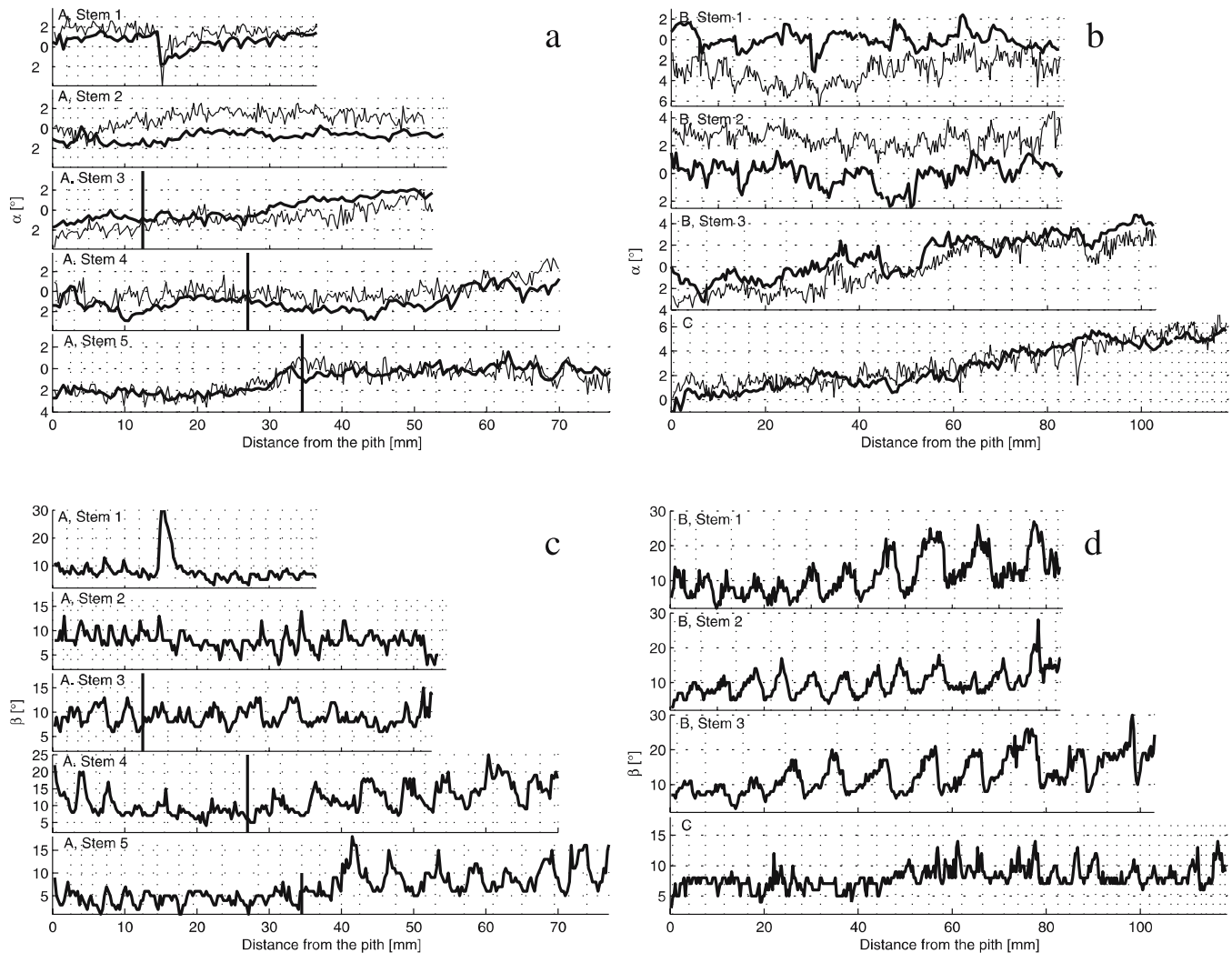


Fig. 2 a,b: Mean fiber orientation in radial-longitudinal plane (α) as a function of the distance from the pith determined with x-ray diffraction (thicker curve) and laser scattering (thinner curve). Dashed vertical lines indicate annual rings. Stems A1 and A2 are control samples and A3–A5 are fertilized. The beginning of the nutrient optimization experiment is denoted by a bold vertical line. Stems B1–B3 are fast grown and C is a stem grown in typical conditions. **c,d:** Mean fiber orientation in tangential-longitudinal plane (β) as a function of the distance from the pith determined with laser scattering. Note: the increased oscillation after the beginning of the fertilization (Stems 2–5, bold vertical line)

Abb. 2 a,b: Mittels Röntgenbeugung (dickere Kurve) und Laserstreuung (dünnere Kurve) bestimmte mittlere Faserneigung in radial-longitudinaler Ebene (α) in Abhängigkeit vom Abstand zur Markröhre. Senkrechte, gestrichelte Linien geben die Jahrringe an. Stämme A1 und A2 sind Referenzproben, A3–A5 sind gedüngt. Der Beginn des Düngungsversuchs ist durch eine dicke, senkrechte Linie dargestellt. Die Stämme B1–B3 sind schnellwüchsig, Stamm C ist unter Normalbedingungen gewachsen. **c,d:** Mittels Laserstreuung bestimmte mittlere Faserneigung in tangential-longitudinaler Ebene (β) in Abhängigkeit vom Abstand zur Markröhre. Auffällig ist die erhöhte Oszillation nach Beginn der Düngung (Stämme 2–5, dicke, senkrechte Linie)

lution was achieved by applying the so called CT-direction method (Ekevad 2004). Unfortunately, this method is sensitive to knots, growth rings and compression wood. Image processing can be used (Lu and Tan 2004), but it is limited only to the surface of the sample. There are also studies on *Eucalyptus nitens* and *Pinus radiata*, where the orientation of fibers was determined using x-ray diffraction (e.g. Evans et al. 1996, Evans 1999). The diameter of the x-ray beam was 0.2 mm, which was also the limiting spatial resolution.

In this paper we study the variation of fiber orientation as a function of age and distance from the pith in stems of Norway spruce grown at different rates in Finland and Sweden. Two independent nondestructive techniques to determine fine changes in the fiber orientation, namely x-ray diffraction (XRD) and laser light scattering, are compared. Both methods are convenient for studying intra-ring variation in the fiber orientation.

2 Materials

The samples were from Norway spruce trees (*Picea abies* [L.] Karst.). Site "A", Flakaliden, is a low fertility site ($H_{100} = 17-19$) in northern Sweden (lat. 64.07 N, long. 19.27 E, alt. 310 m from sea level). The nutrition optimization experiment at Flakaliden was started when the trees were 12 years old and continued until the trees were cut (Linder 1995). Samples A1 and A2 were control samples and samples A3–A5 were from fertilized trees. The increase in volume growth was almost four times that of the control plants grown in the same area (Mäkinen et al. 2001). The mean microfibril angles were reported by Peura et al. (2005), and fiber characteristics and density by (Mäkinen et al. 2002a, Mäkinen et al. 2002b).

Site "B" was an experimental site of the Finnish Forest Research Institute at Nurmijärvi, Finland (lat. 60.30 N, long 24.42 E, alt. 100 m from sea level), which was former agricultural land with a high fertility level ($H_{100} \sim 34$). The Norway spruce stems were cutting clones of a fast-grown mother tree, which was a hybrid between a tree from Pieksämäki, Finland (lat. 62.18 N, long 27.10 E, alt. 100 m from sea level) and a tree from Schilbach, Germany (lat. 50.25 N, long. 12.30 E, alt. 600 m from sea level). Samples were taken from these 20-year old trees. In addition, one sample, site "C", was from Ruotsinkylä (lat. 60.21 N, long. 24.59 E, alt. 49 m from sea level), Finland, which is a medium fertility site ($H_{100} \sim 24$). The average ring widths were 6 mm for site "B" and 2 mm for "C". The mean microfibril angles and fiber characteristics were reported by (Sarén et al. 2001, Sarén et al. 2004).

Wood discs were cut at breast height, 1.3 m. From each disc, wood sections with a thickness of 1.0 mm in the tangential direction were cut in the transverse direction from the pith to the bark using a double circular saw. The "B" samples were cut into several smaller pieces during previous x-ray experiments. Therefore, the same samples could not be used for laser light studies and laser measurements were performed from a wood block left out of preparation of the x-ray samples. The thickness of these blocks was 20 mm in the tangential direction. Di-

stances between the samples for these two measurements were about 1.5 mm in the tangential direction of the stem. Since it is known that surface roughness has an effect on optical scattering in wood (Shen et al. 2000), all the samples were cut with the same machine.

During the sample preparation the exact longitudinal direction of the stem can easily be lost. The errors made during sample preparation, mainly in cutting discs from the trees, can be a few degrees. Therefore, there may be some offset in values of the angle α (Fig. 2a and b).

3 Methods

3.1 X-ray diffraction

The diffraction pattern of a wood sample contains information on both the helical orientation of cellulose microfibrils in the cell walls and the mean orientation of the fibers in the sample (the angle α , Fig. 1). If the microfibril angle distribution is the same in opposite cell walls the diffraction pattern is symmetric with respect to the cell axis. If the cells are not parallel to the radial-longitudinal surface of the sample (Fig. 1 $\beta \neq 0$), the shapes of the peaks of the intensity curve become asymmetric but the diffraction pattern remains symmetric with respect to the cell axis (Klug and Alexander 1974). This may affect the determination of the mean microfibril angle, but not the orientation.

The XRD measurements were performed with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$) generated using a rotating anode X-ray source and monochromatized and focused using Göbel-mirrors (Sarén et al. 2004). The diameter of the beam at the position of the sample was about 0.5 mm and the sample-to-detector distance was 102.0 mm. The incoming X-ray beam was perpendicular to radial cell walls and the scattered intensity was measured using an area detector (Hi-Star, Bruker AXS) positioned perpendicular to the incoming beam. Wood sections with a thickness of 1.0 mm in the tangential direction were moved with a motorized sample holder with a step of 0.5 mm in the radial direction from the pith towards the bark. The measurement time for each frame (512×512 pixels) was 900 seconds. Each frame was spatially and flood field corrected using GADDS 3329 program (Bruker AXS, Karlsruhe, Germany). Owing to the good angular homogeneity of the background, no background subtraction was done.

The cellulose reflection 200, indexed according to Sugiyama et al. (1990), was used in the analysis. The intensity curves as a function of the polar angle φ were obtained from each frame by integrating the intensity over a band of 21.6° – 23.6° in 2θ -scale. The mean microfibril angle was determined by fitting a model to this intensity curve (Sarén et al. 2004). The orientation of the fibers in radial-longitudinal plane (Fig. 1, the angle α) was determined from the position of the peaks in φ -scale compared to the orientation of the sample. The accuracy of the mean α was estimated to be $\pm 2^\circ$. The frames from the same annual ring could be easily identified on the basis of the intensity curves for the cellulose reflection 200. The positions of the annual rings were confirmed using digital images of the samples.

3.2 Laser light scattering

The equipment was a prototype device consisting of a He-Ne laser, a charge-coupled device (CCD) camera, and a computer. Approximately 5% of the laser light with the wave length of 632.8 nm is transmitted through these samples, about 45% is absorbed, and 50% is reflected. The depth of penetration of the beam is about 3 mm. The width of the beam on the surface of the sample was 0.5 mm. Seven intensity ovals were determined in an image taken by the CCD camera using principal component analysis (PCA) (Simonaho et al. 2004). Both absorbing and reflecting masks behind the sample were tested to eliminate the effect of the transmitted light for thin samples (A1–A5 and C). However, no differences in the determined angles were found.

The surface of the sample was considered as a cylindrical reflecting surface. The fiber is considered as an anisotropic absorbing tube; light propagated across wood cells walls was attenuated more than light propagated along fiber walls in longitudinal direction of the fibers. Part of the light was propagated back to the surface, thus forming a quasi-elliptical intensity pattern (Shen et al. 2000, Zhou and Shen 2003). The major axis of this ellipse was parallel to the longitudinal direction of the fibers (Fig. 1, the angle α). The accuracy of the angle was estimated to be about 1°.

The angle β between the fiber axis and the sample surface (Fig. 1) was calculated from the ratio between the minor and major axes of the quasi-elliptical intensity pattern (Nyström 2002, Nyström 2003, Simonaho et al. 2004). Therefore, the values of the angle β are always positive. The accuracy of the angle was estimated to be about 2°.

4 Results and discussion

The angle α , which gives the fiber orientation in radial-longitudinal plane, was determined using both x-ray and laser light scattering. The same trend as a function of the distance from the pith was observed (Fig. 2a and b). The offset between the curves is due to differences in the positioning of the sample. The angle α increased almost linearly from the pith to the bark for most of the samples (A2–A5, B3, C) and there was no clear trend from the pith to the bark in B1 and B2 (fast-grown clonal trees, fertile site). There was an abrupt change in the angle α at the 15 mm from the pith (8th annual ring) of the sample A1 (control sample for fertilization experiment), which could be also seen in the angle β (Fig. 2c). This was attributed to a visible defect, false annual ring, in the sample. In most of the samples (A2–A5, B3, and C) the angle α near the pith was slightly negative. In the radial-longitudinal plane upper tips of fibers were thus tilted towards the pith and the tilt angle increased gradually towards the bark.

The variation of the angle α was determined separately for earlywood, latewood, and the whole annual ring. For earlywood we used the definition first 70% of the thickness of the annual ring because other parameters were determined accordingly. The angle α varied both within and between annual rings more in the samples B than in the other samples. The variation of the angle α

within an annual ring was greatest in earlywood of the samples B. This is attributed to the special genetic background of the samples B. The range between the largest and smallest angle α was smaller in earlywood than in latewood.

The beginning of the fertilization did not change the angle α markedly in samples A3–A5 (Fig. 2a). However, the overall variation of the angle α in the same distance range was smaller in narrow than in broad annual rings.

The spiral grain angle β was determined using laser light scattering. For samples from normally grown stems (A1, A2, C) the angle β did not vary much with the distance from the pith (Fig. 2c and d). In these samples it was between 5–14 degrees, with an exception at the 15 mm from the pith (8th annual ring) of the sample A1. The values are higher than reported previously for similar material: according to Gjerdrum et al. (2002) the average spiral grain angle decreased from 2 to –1 degrees, according to Kliger (2001) from 2.1 to 0.9 degrees, and according to Hannrup et al. (2004) from 4 to 1 from about 10 mm from the pith to 90 mm from the pith. Gjerdrum et al. (2002) reported that the overall trend for the grain angle as a function of the distance from the pith to the bark is linear in Norway spruce, and both the value at the pith and the slope of β varied substantially for each stem.

The high values of the angle β are attributed to the measurement setup. The relationship between the eccentricity and the angle β was calibrated using a tangential surface of a block of wood, which was further cut in known angle with respect to the fiber axis (Simonaho et al. 2004). The suitability of the same calibration for both normally and fast grown trees and for latewood and earlywood has not been studied. The samples studied in this work were cut in the radial direction. Therefore, bordered pits that are only in radial cell walls may cause an increase of the eccentricity and the angle β . This may explain an offset in the values of the angle β ; however, changes in the angle β do not depend on calibration.

In the samples (B1–B3) from fast grown trees and in fertilized annual rings of the samples A the angle β was 0.7 degrees higher in earlywood than in latewood. For other samples the average value was 0.2 degrees lower in earlywood. This is in agreement with Wobst et al. (1994), who reported that the difference between earlywood and latewood was 0.58 degrees in ash and 0.20 degrees in Douglas fir.

In the case of the samples A3–A5 and B1–B3, where the growth rate was high, the angle β oscillated considerably. Fertilization was reported to increase ethylene evolution and to maintain high left-handed spiral grain compared to slower grown trees (Eklund et al. 2003). In samples A the amplitude of the oscillation of the angle β increased considerably after the fertilization was started. Interestingly, the period of the oscillation was not exactly the same as the thickness of the corresponding annual ring. Despite that, ranges of variations were higher in latewood than in earlywood (Fig. 3). It has been shown that the formation of spiral grain is genetically controlled (Harris 1989, Hannrup et al. 2004) and the differences in the orientation of fibers between samples from the fertilized stems A3–A5 and B1–B3 are likely genetic.

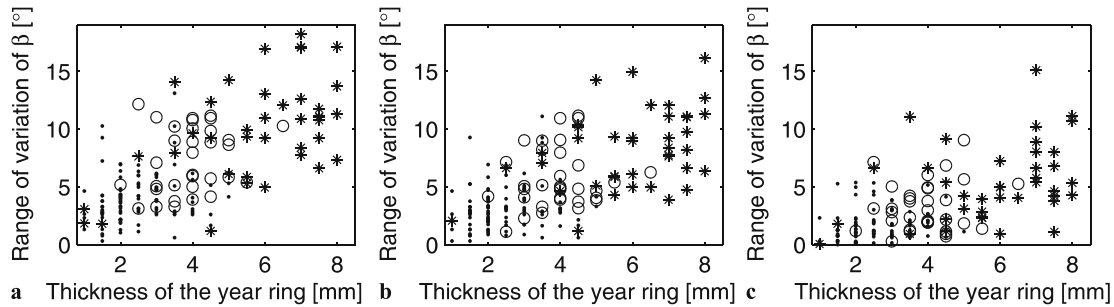


Fig. 3 The ranges of variation of (β) as a function of the thickness of the annual ring for the whole annual ring (A), earlywood (B), and latewood (C). Pearson correlations were 0.645, 0.624, 0.628 for A, B, and C, respectively. The control samples (A1–A2, C and A3–A5 before fertilization) are marked with (.), samples after fertilization (A3–A5) with (o), and fast-grown samples (B1–B3) with (*)

Abb. 3 Variationsbreite von (β) in Abhängigkeit von der Jahrringbreite im (A) gesamten Jahrring, (B) Frühholz und (C) Spätholz. Pearson Korrelationskoeffizienten ergaben 0,645, 0,624, 0,628 für A, B und C. Die Referenzproben (A1–A2, C und A3–A5 vor Düngung) sind mit (.) markiert, Proben nach Düngung (A3–A5) mit (o) und schnellwüchsige Proben (B1–B3) mit (*)

The mean microfibril angle and the circularity index of the lumen are among those structural parameters that are different near the pith (say, first 10 annual rings) and in mature wood of Norway spruce (Sarén et al. 2001, Sarén et al. 2004). Also the length and the diameter of the fibers change quite drastically in juvenile wood, but both change smoothly in mature wood. Since the angles α and β (Figs. 2, 3 and 4) do not show such behavior, we conclude that they do not depend linearly on the microfibril angle, the circularity index, the length or the diameter of the fibers. As an example, Fig. 4 shows the microfibril angle of early and latewood and the circularity index of the earlywood for sample A2. The mean microfibril angle is higher in earlywood than in latewood, with an exception of the first annual ring. On the basis of the microfibril angle and the circularity index, there is compression wood at the distance of 40–50 mm from the pith. This was confirmed by visual inspection of the cross-section. The sample C contained also compression wood at the distance of 40–66.5 mm (the annual rings 11–17). Compression wood did not change either α or β .

Some of the structural parameters, e.g. the cell wall thickness, the radial diameter of the fibers, and the fiber length, are different for early and latewood and thus show an oscillatory behavior as a function of the distance from the pith (e.g. Chalk and Ortiz 1961, Sarén et al. 2001, Mäkinen et al. 2002a). In the angle α , however, there are no statistically significant oscillations. The period of the oscillation of the angle β is not exactly the same as the thickness of the annual ring, so no evident correlation between the fiber orientation and these parameters could be found.

The thickness of the annual ring was found to be the only parameter investigated, which was correlated to the range or mean value of the angle β (Fig. 3). This is in agreement with results of the thinning experiment of Pape (1999), who found correlation between the ring width and spiral grain, especially when thinning was heavy.

To summarize, there was no clear relationship between fiber orientation and fiber dimensions. This is in agreement with Eklund et al. (2003), who found out that there is correlation between the spiral grain angle and the number of the fibers for-

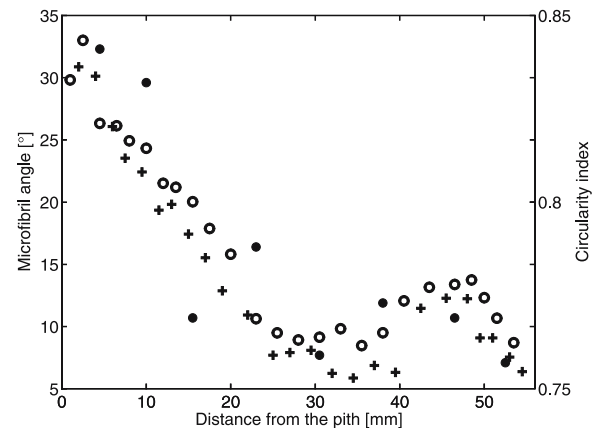


Fig. 4 The mean microfibril angle in earlywood (o) and latewood (+) and the circularity index in earlywood (●) of the sample A2

Abb. 4 Mittlerer Mikrofibrillenwinkel im Frühholz (o) und Spätholz (+) sowie der Rundheitsindex im Frühholz (●) der Probe A2

med in an annual ring, but no correlation between the spiral grain angle and the radial diameter of the fibers.

5 Conclusion

In this work we showed that the angle α describing the orientation of fibers in radial-longitudinal plane determined using laser light and x-ray scattering are in good agreement. The angle α increased gradually from the pith to the bark in almost all samples. In fast grown samples there were more variations in α both within and between annual rings. The presence of compression wood did not affect fiber orientation remarkably.

The spiral grain angle (β), determined using laser scattering, oscillated. The local period of the oscillation did not correspond with annual ring width. The amplitude of the oscillation was higher in fast grown samples, which suggests that fast growth may promote spiral grain, but more measurements are needed to verify this.

It was possible to distinguish between samples with high and normal growth rate using laser light scattering. This technique could be useful for screening sawn timber with high twisting probability. It is also possible to determine fiber orientation (α) from the surface of plywood or rotary-cut veneer. In general, such results can be useful for predicting and ensuring properties of manufactured materials.

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